LLNL-TR-637818

LAWRENCE LIVERMORE NATIONAL LABORATORY

Integrated Data Collection Analysis (IDCA) Program - Bullseye Smokeless Powder

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May 30, 2013

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This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Integrated Data Collection Analysis (IDCA) Program —Bullseye® Smokeless Powder

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ABSTRACT

The Integrated Data Collection Analysis (IDCA) program is conducting a proficiency study for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the results for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of Bullseye® smokeless powder (Gunpowder). The participants found the Gunpowder: 1) to have a range of sensitivity to impact, from less than RDX to almost as sensitive as PETN, 2) to be moderately sensitive to BAM and ABL friction, 3) have a range for ESD, from insensitive to more sensitive than PETN, and 4) to have thermal sensitivity about the same as PETN and RDX.

This effort, funded by the Department of Homeland Security (DHS), is putting the issues of safe handling of these materials in perspective with standard military explosives. The study is adding SSST testing results for a broad suite of different HMEs to the literature. Ultimately the study has the potential to suggest new guidelines and methods and possibly establish the SSST testing accuracies needed when developing safe handling practices for HMEs. Each participating testing laboratory uses identical test materials and preparation methods. Note, however, the test procedures differ among the laboratories. The testing performers involved are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), Sandia National Laboratories (SNL), and Air Force Research Laboratory (AFRL/RXQL). These tests are conducted as a proficiency study in order to establish some consistency in test protocols, procedures, and experiments and to compare results when these testing variables cannot be made consistent.

Keywords: Small-scale safety testing, proficiency test, impact-, friction-, spark discharge-, thermal testing, round-robin test, safety testing protocols, HME, RDX, potassium perchlorate, potassium chlorate, sodium chlorate, sugar, dodecane, PETN, carbon, ammonium nitrate, Gunpowder, Bullseye® smokeless powder.

IDCA Explosives Safety Testing of Homemade Explosives
Integrated Data Collection Analysis Program

1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory Small-Scale Safety and Thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives[1]. The materials for the Proficiency Test have been selected because their properties invoke challenging experimental issues when testing HMEs. Many of these challenges are not normally encountered with military type explosives. To a large extent, the issues are centered on the physical forms and stability of the improvised materials.

Often, HMEs are formed by mixing oxidizer and fuel precursor materials, and typically, the mixture precursors are combined shortly before use. The challenges to produce a standardized inter-laboratory sample are primarily associated with mixing and sampling. For solid-solid mixtures, the challenges primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uniform composition—particle size and dryness often being a factor—as well as taking a representative sample. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around the ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products.

Table 1. Materials for IDCA Proficiency study

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	Powder mixture
Potassium perchlorate	Charcoal	Powder mixture
Potassium perchlorate	Dodecane[1]	Wet powder
Potassium chlorate	Dodecane[1]	Wet powder
Potassium chlorate as received	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Potassium chlorate -100 mesh[3]	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Sodium chlorate	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Ammonium nitrate		Powder
Bullseye® smokeless powder[4]		Powder
Ammonium nitrate	Bullseye® smokeless powder[4]	Powder mixture
Urea nitrate	Aluminum	Powder mixture
Urea nitrate	Aluminum, sulfur	Powder mixture
Hydrogen peroxide 70%	Cumin	Viscous paste
Hydrogen peroxide 90%	Nitromethane	Miscible liquid
Hydrogen peroxide 70%	Flour (chapatti)	Sticky paste
Hydrogen peroxide 70%	Glycerine	Miscible liquid
HMX Grade B		Powder
RDX Class 5 Type II		Powder (standard)
PETN Class 4		Powder (standard)

1. Simulates diesel fuel; 2. Contains 3 wt. % cornstarch; 3. Sieved to pass 100 mesh; 4. Alliant Bullseye® smokeless pistol gunpowder.

The IDCA has chosen several formulations to test that present these challenges. Table 1 shows the materials selected for the Proficiency Test and the Description column describes the form of the resulting material. Details of the results from the Proficiency Test for the materials examined are documented in IDCA Analysis Reports—RDX first testing[2], RDX second testing[3], RDX testing comparison[4], $KClO_3$/sugar (separated with a 100 mesh sieve)[5], $KClO_3$/sugar (as received)[6], $KClO_3$/Dodecane[7], $KClO_4$/Dodecane[8], $KClO_4$/Al[9], $KClO_4$/Carbon[10], $NaClO_3$/sugar[11], PETN[12], Methods[13], and Ammonium Nitrate[14].

Evaluation of the results of SSST testing of unknown materials, such as the HMEs in Table 1, is generally done as a relative process, where an understood standard is tested alongside the HME. In many cases, the standard employed is PETN or RDX. The standard is obtained in a high purity, narrow particle size range, and measured frequently. The performance of the standard is well documented on the same equipment (at the testing laboratory), and is used as the benchmark. The sensitivity to external stimuli and reactivity of the HME (or any energetic material) are then evaluated relative to the standard.

Most of the results from SSST testing of HMEs are not analyzed any further than this. The results are then considered in-house. This approach has worked very well for military explosives and has been a validated method for developing safe handling practices. However, there has never been a validation of this method for HMEs. Although it is generally recognized that these SSST practices are acceptable for HME testing, it must always be kept in mind that HMEs have different compositional qualities and reactivities than conventional military explosives.

The IDCA is evaluating SSST testing methods as applied to HMEs. In addition, the IDCA is attempting to understand, at least in part, the laboratory-to-laboratory variation that is expected when examining HMEs. The IDCA team has taken several steps to make this inter-laboratory data comparison easier to analyze. Each participating laboratory uses materials from the same batches and follows the same procedures for synthesis, formulation, and preparation. In addition, although the Proficiency test allows for laboratory-to-laboratory testing differences, efforts have been made to align the SSST testing equipment configurations and procedures to be as similar as possible, without significantly compromising the standard conditions under which each laboratory routinely conducts their testing.

The first and basic step in the Proficiency test is to have representative data on a standard material to allow for basic performance comparisons. Table 1 includes some standard military materials. Class 5 Type II RDX was chosen as the primary standard, and Class 4 PETN was chosen as a secondary material. These materials have been tested in triplicate and RDX was tested throughout the IDCA Proficiency Test.

The subject of this report, Gunpowder, is the ninth HME tested in the Proficiency Test and is one of set of three related tests—AN, Gunpowder and AN/Gunpowder mixture. Gunpowder was selected because it is a solid component that when combined with AN, will again demonstrate the challenges of SSST testing of two fine solids mixed together. The Gunpowder chosen is Bullseye® smokeless powder (not a product endorsement), a double-base powder containing nitroglycerin and nitrocellulose. The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), and Indian Head Division, Naval Surface Warfare Center, (NSWC IHD).

2 EXPERIMENTAL

General information. All samples were prepared according to IDCA methods on drying and mixing procedures[15,16]. However, the gunpowder was not dried before testing. The Bullseye® smokeless powder was from Alliant Powder Company. The composition (according to the manufacturer) is NG 40%, NC 58%, Ethyl Centralite (stabilizer) 1%, modifier and graphite 1%. The material was packaged in May, 2003 (the manufacturer suggested the stabilizer level be checked once every 5 years). The average particle properties were measured by laser diffraction light scattering method using Microtracs Model FRA9200.

Testing conditions. Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the Gunpowder. SSST testing data for the individual participants was obtained from the following IDCA Data Reports: Small Scale Safety Test Report for Smokeless Gunpowder (LLNL)[17], 50188 J Bullseye® Smokeless Pistol Gunpowder (LANL)[18], and Bullseye® (IHD)[19].

Table 2. Summary of conditions for the analysis of RDX (All = LANL, LLNL, IHD)

Impact Testing

1. Sample size—LLNL, IHD, 35 ± 2 mg; LANL, 35 or 40 ± 2 mg
2. Preparation of samples—All, as received
3. Sample form—All, loose powder
4. Powder sample configuration—All, conical pile
5. Apparatus—LANL, LLNL, IHD, Type 12*
6. Sandpaper—All, 180-grit garnet dry
7. Sandpaper size—LLNL, IHD, 1 inch square; LANL, 1.25 inch diameter disk dimpled
8. Drop hammer weight—All, 2.5 kg
9. Striker weight—LLNL, IHD, 2.5 kg; LANL, 0.8 kg
10. Positive detection—LANL, LLNL, microphones with electronic interpretation as well as observation; IHD, observation
11. Data analysis—All, modified Bruceton; LANL Neyer also

Friction analysis

1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, as received
3. Sample form—All, powder
4. Sample configuration—All, small circle form
5. Apparatus—LANL, LLNL, IHD, BAM; IHD, ABL
6. Positive detection—All, by observation
7. Room Lights—LANL on; and LLNL off; IHD, BAM on, ABL off
8. Data analysis—LLNL modified Bruceton (log-scale spacing) and TIL; LANL and IHD, modified Bruceton (linear spacing) and TIL

ESD

1. Sample size—All ~5 mg, but not weighed
2. Preparation of samples—All, as received
3. Sample form—All, powder
4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD, none
5. Sample configuration—All, cover the bottom of sample holder
6. Apparatus—LANL, IHD, ABL; LLNL, custom built*
7. Positive detection—All, observation
8. Data analysis methods—All, TIL

Differential Scanning Calorimetry

1. Sample size—All ~ <1 mg
2. Preparation of samples—All, as received
3. Sample holder—All, pinhole; LLNL, TA sealed
4. Scan rate—All, 10°C/min
5. Range—All, 40 to 400°C+
6. Sample holder hole size—LANL, IHD, 75 μm; LLNL, 50 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920; IHD, TA Instruments Q1000*

Footnotes: *Test apparatus, *Impact:* LANL, LLNL, IHD—ERL Type 12 Drop Weight Sensitivity Apparatus, AFRL, SNL— MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD, SNL—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark:* LLNL, LANL, IHD, AFRL, SNL—ABL Electrostatic Discharge Apparatus, LLNL—custom-built Electrostatic Discharge Apparatus; *Differential Scanning Calorimetry:* LANL—TA Instruments Q1000, Q2000, LLNL—TA Instruments 2910, 2920, Setaram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

3 RESULTS

3.1 Gunpowder

In this proficiency test, all testing participants are required to use materials from the same batch, and mixtures are to be prepared by the same methods. However, the actual testing procedures can be different. These differences are described in the IDCA Analysis Report on method comparisons[13], which compares procedures by each testing category. LANL, LLNL and IHD participated in this testing.

3.2 Particle Size Distribution of Gunpowder

Figure 1 shows the particle size distribution of the Gunpowder performed by laser light scattering[20]. The distribution extends from 500 to 1000 μm (10% 530 μm, 95% 970 μm). The average particle size is 753 ± 153 μm.

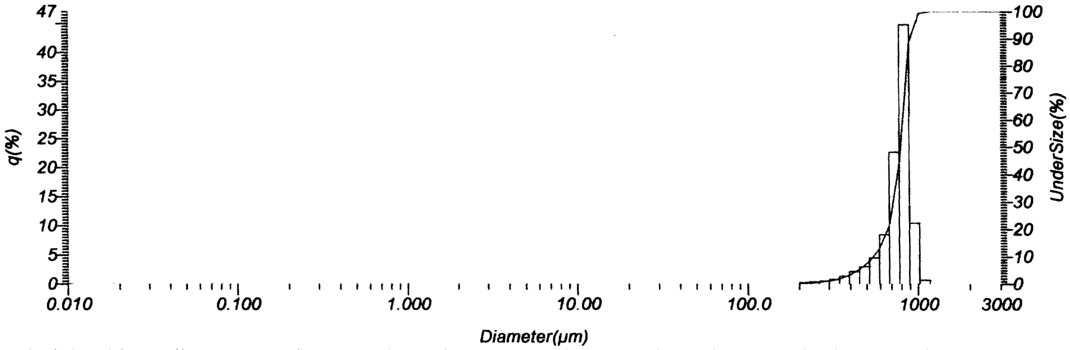

Figure 1. Microtracs laser light scattering particle size distribution for Bullseye® Gunpowder

3.3 Impact testing results for Gunpowder

Table 3 shows the results of impact testing of Gunpowder performed by LANL, LLNL and IHD. Differences in the testing procedures are shown in Table 2, and the notable differences are the amount of sample, and the methods for detection of a positive test. All participants performed data analysis by a modified Bruceton method[21,22]. All participants found the Gunpowder to have different sensitivity in impact testing. The average values of the DH_{50} for each participant are LLNL, 54.4 ± 4.9 cm; LANL, 20.7 ± 0.99 cm; IHD, 12.3 ± 0.58 cm. Total average is 29.1 ± 19.6 cm, demonstrating the very large spread in values.

Table 3. Impact testing results for Gunpowder

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (180)	11/08/10	23.9	22	48.7	4.72	0.042
LLNL (180)	11/12/10	23.9	15	56.0	2.97	0.023
LLNL (180)	11/17/10	23.9	18	58.0	4.70	0.035
LANL (180)	1/04/11	20.3	< 10	21.2	2.10	0.043
LANL (180)	1/04/11	20.3	< 10	19.6	1.99	0.044
LANL (180)	1/05/11	21.4	< 10	21.4	1.43	0.029
IHD (180)	12/10/10	23	43	13	1.80	0.06
IHD (180)	12/10/10	23	44	12	2.79	0.10
IHD (180)	12/13/10	20	51	12	1.94	0.07

1. Value in parenthesis is grit size of sandpaper (180 is 180-grit garnet dry); 2. Relative humidity; 3. DH_{50}, in cm, is by a modified Bruceton method, height for 50% probability of reaction; 4. Standard deviation.

Table 4. Impact testing results for Gunpowder (Neyer or D-Optimal Method)

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (180)	1/04/11	20.3	< 10	19.7	1.6	0.035
LANL (180)	1/04/11	20.2	< 10	22.1	1.6	0.031
LANL (180)	1/05/11	20.0	< 10	21.1	1.1	0.023

1. Value in parenthesis is grit size of sandpaper (180 is 180-grit garnet dry); 2. Relative humidity; 3. DH_{50}, in cm, is by the Neyer D-Optimal method, height for 50% probability of reaction; 4. Standard deviation.

Table 4 shows the impact test results from LANL using the Neyer or D-Optimal method[23]. The LANL average value for DH_{50} is 21.0 ± 1.2 cm, similar to the average value for DH_{50} determined by the Bruceton method.

3.4 Friction testing results for Gunpowder

Table 5 shows the BAM Friction testing of Gunpowder performed by LLNL, LANL, and IHD. The difference in testing procedures by the three laboratories is shown in Table 2, and the notable differences are in the methods for positive detection. All participants performed data analysis using the threshold initiation level method (TIL)[24]. LANL and LLNL also used a modified Bruceton method[21,22] and IHD did not use the Bruceton method because their data did not meet Bruceton criteria. Table 5 shows that data on the sensitivity of the mixture varies depending upon on which participant. The average values for F_{50}, in kg are: LLNL 20.7 ± 1.8 and LANL 9.3 ± 0.6. The TIL values follow a trend. The order and average TIL values, in kg, are: LLNL 16.4 > IHD 13.8 > LANL 5.6.

Table 5. BAM Friction Testing results for Gunpowder

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4,5]	s, kg[6]	s, log unit[6]
LLNL	11/05/10	23.9	32	0/10 @ 18.0	1/10 @ 19.2	22.7	4.1	0.078
LLNL	11/11/10	23.9	18	0/10 @ 16.8	1/10 @ 17.4	20.2	3.3	0.070
LLNL	2/11/11	23.9	21	0/10 @ 14.4	1/10 @ 16.0	19.1	2.4	0.054
LANL	1/03/11	20.2	< 10	NA[7]	NA[7]	8.8	1.7	0.085
LANL	1/04/11	20.0	< 10	NA[7]	NA[7]	10.0	3.1	0.139
LANL	1/04/11	19.9	< 10	NA[7]	NA[7]	9.2	2.9	0.141
LANL	1/03/11	20.1	< 10	0/10 @ 4.8	1/6 @ 7.2	NA[8]	NA[8]	NA[8]
LANL	1/04/11	19.8	< 10	0/10 @ 7.2	1/4 @ 9.6	NA[8]	NA[8]	NA[8]
LANL	1/04/11	19.8	< 10	0/8 @ 4.8	1/8 @ 7.2	NA[8]	NA[8]	NA[8]
IHD	1/10/11	22	41	NA[7]	NA[7]	> 36	NA[9]	NA[9]
IHD	1/5/11	24	40	0/10 @ 14.7	1/3 @ 16.5	NA[8]	NA[8]	NA[8]
IHD	1/5/11	24	40	0/10 @ 12.2	1/2 @ 14.7	NA[8]	NA[8]	NA[8]
IHD	1/5/11	24	42	0/10 @ 14.7	1/8 @ 16.5	NA[8]	NA[8]	NA[8]

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 5. LLNL uses log spacing and LANL uses liner spacing for the Bruceton up and down method experimentation and data analysis 6. Standard deviation; 7. Not applicable, separate measurement performed for TIL; 8. Not applicable, separate measurements performed for modified Bruceton analysis; 9. Not applicable, outside the range of the Bruceton analysis.

Table 6 shows the ABL Friction testing of Gunpowder performed by IHD. LANL did not have the system in routine performance at the time. LLNL and SNL do not have ABL Friction testing equipment. IHD performed data analysis using a modified Bruceton analysis[21,22]. The F_{50} data show that the Gunpowder has some friction sensitivity.

Table 6. ABL Friction testing results for Gunpowder

Lab	Test Date	T, °C	RH, %[1]	TIL, psig/fps[2,3]	TIL, psig/fps[4]	F_{50}, psig/fps[5]	s, psig[6]	s, log unit[6]
IHD	12/22/10	25	45	NA[7]	NA[7]	310/8	94	0.13
IHD	12/22/10	25	45	NA[7]	NA[7]	304/8	49	0.07
IHD	12/22/10	25	44	NA[7]	NA[7]	335/8	94	0.12

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 4. Next level where positive initiation is detected; 5. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% probability of reaction; 6. Standard deviation; 7. Not applicable, TIL measurement not performed.

3.5 Electrostatic discharge testing results for Gunpowder

Electrostatic Discharge (ESD) testing of Gunpowder was performed by LLNL, LANL and IHD. Table 7 shows the results. Differences in the testing procedures are shown in Table 2, and the notable differences are the use of tape covering the sample. In addition, LLNL uses a custom built ESD system with a 510-Ω resistor in line to simulate a human body, making a direct comparison of the data from LLNL with data generated by the other participants challenging. All participants performed data analysis using the threshold initiation level method (TIL)[24].

Table 7. Electrostatic discharge testing results for Gunpowder

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	11/05/10	23.9	32	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	11/11/10	23.9	18	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	11/15/10	24.4	23	0/10 @ 1.0	0/10 @ 1.0
LANL[5]	1/03/11	20.0	< 10	0/20 @ 0.025	3/4 @ 0.0625
LANL[5]	1/04/11	20.4	< 10	0/20 @ 0.025	1/2 @ 0.0625
LANL[5]	1/04/11	20.4	< 10	0/20 @ 0.025	2/3 @ 0.0625
IHD[5]	12/09/10	22	40	0/20 @ 0.1625	1/8 @ 0.326
IHD[5]	12/09/10	21	40	0/20 @ 0.1625	1/1 @ 0.326
IHD[5]	12/09/10	21	40	0/20 @ 0.1625	1/1 @ 0.326

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. LLNL used a custom built ESD with a 510-Ω resistor in the discharge unit to mimic the human body. 5. ABL ESD equipment.

For TIL, IHD found the material to be the most stable, while LANL found it less sensitive. The LLNL values using the custom built system show a material with no sensitivity.

3.6 Thermal testing (DSC) results for Gunpowder

Differential Scanning Calorimetry (DSC) was performed on Gunpowder by LLNL, LANL and IHD. All participating laboratories used different versions of the DSC by TA Instruments. Table 8 shows the data. Results were obtained at a 10°C/min heating rate.

Table 8. Differential Scanning Calorimetry results for Gunpowder, 10°C/min heating rate

Lab	Test Date	Exothermic, onset/maximum, °C (ΔH, J/g)[1,2]
LLNL[3]	11/18/10	~ 145/192.0 (3458)[4]
LLNL[3]	11/18/10	~ 145/204.3 (3491)[4]
LLNL[3]	11/18/10	~ 155/200.7 (2231)[4]
LLNL[5]	11/19/10	~ 155/203.4 (1964)[4]
LLNL[5]	11/19/10	~ 150/201.0 (2044)[4]
LLNL[5]	11/19/10	~ 155/202.7 (1942)[4]
LANL[5]	1/3/11	154.4/199.3 (2089)[6]
LANL[5]	1/5/11	153.0/200.6 (1964)
LANL[5]	1/7/11	157.4/200.0 (1836)
IHD[5]	3/23/11	~ 150/199.9 (2226)[4]
IHD[5]	3/23/11	~ 150/199.3 (2149)[4]
IHD[5]	3/23/11	~ 150/198.9 (2184)[4]

1. Exothermic = ΔH positive; 2. Maximum = maximum temperature of transition, T_{max}; 3. Hermetically sealed sample holder; 4. Onset temperature estimated on hard copy; 5. pinhole sample holder; 6. Unexplained extremely sharp transition at 190°C.

Table 8 shows the DSC data taken with a pinhole or a hermetically seal sample holder. All participants measured one, fairly broad, exothermic feature. For LLNL and IHD, the onset of this exothermic feature was only approximated, while LANL notated the value. For the pinhole sample holder, the data looks almost identical when comparing the contributions from each participant. The average for the T_{max} is 200.6 ± 1.6 °C and the ΔH is 2044 ± 129 J/g (a relative variation of about 6 %). The data taken with the hermetically sealed sample holder fits into this range, although the exothermic feature enthalpy is somewhat higher.

There have been many studies on the thermal decomposition of double-base gunpowder and the components thereof. The T_{max} and ΔH_{exo} values in Table 8 agree for the most part with studies on a wide variety of differing compositions of Gunpowder and the major components, nitrocellulose and nitroglycerin[25-30].

4 DISCUSSION

Table 9. Average Comparison values

	LLNL	LANL	IHD
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
Gunpowder[2-4]	54.2	20.7	12.3
RDX Type II Class 5[3,5]	22.6	20.9	19.7
PETN[3,6]	8.3	8.0	9.3
BAM Friction Testing[7,8]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
Gunpowder[9,10]	16.4; 20.7	5.6; 9.3	13.8; NA[11]
RDX Type II Class 5[5]	19.2; 25.1	19.2; 20.8	15.5; ND[12]
PETN[6]	6.4; 10.5	4.9; 8.5	4.3; 6.9
ABL Friction Testing[13-16]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
Gunpowder[17,18]	ND[12]; ND[12]	ND[12]; ND[12]	ND[12]; 317
RDX Type II Class 5[5]	ND[12]; ND[12]	ND[12]; ND[12]	74; 154
PETN[6]	ND[12]; ND[12]	ND[12]; ND[12]	7.7; 42
Electrostatic Discharge[19]	TIL, Joules	TIL, Joules	TIL, Joules
Gunpowder[20,21]	0/10 @ 1.0[22]	0/20 @ 0.0250[23]	0/20 @ 0.1625[23]
RDX Type II Class 5[5]	0/10 @ 1.0[22]	0/20 @ 0.0250[23]	0/20 @ 0.095[23]
PETN[6]	0/10 @ 0.033[23]	0/20 @ 0.025[23]	0/20 @ 0.219[23]

1. DH_{50}, in cm, is by a modified Bruceton method, load for 50% probability of reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 15-22), LANL (20.3-21.4; <10), IHD (20-23; 43-51); 3. 180-grit sandpaper; 4. Average of three measurements from Table 3; 5. From reference 4; 6. From reference 12; 7. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 8. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 9. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 18-32), LANL (19.8-20.2; <10), IHD (22-24; 40-42); 10. Average of three measurements from Table 5; 11. Outside the range of the Bruceton analysis; 12. ND = Not determined; 13. LLNL and LANL did not perform measurements; 14. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 15. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% probability of reaction; 16. Measurements performed at 8 fps; 17. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (25; 44-45); 18. Average of three measurements from Table 6; 19. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 20. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9-24.4; 18-32), LANL (20.0-20.4; <10), IHD (21-22; 40); 21. Average of three measurements from Table 7; 22. LLNL has 510-Ω resistor in circuit; 23. ABL ESD apparatus.

Table 9 shows the average values for the data for Gunpowder from each participant and compares it to corresponding data for standards, RDX Type II Class 5 and PETN Class 4 done previously. The data for RDX comes from the evaluation of all of the RDX examinations as part of this Proficiency Test[4], and the data for PETN comes from the examination of PETN Class 4 as part of this Proficiency Test[12].

4.1 Comparison of participating laboratory testing of Gunpowder

Impact sensitivity. All the data in Table 9 for the Gunpowder is from testing using 180-grit garnet sandpaper in the drop hammer experiment. All three participants show the Gunpowder has distinctively different sensitivities. LLNL found the lowest sensitivity and IHD found the highest. LANL analysis by the Neyer method yielded about the same sensitivity as the LANL values using Bruceton analysis.

Friction sensitivity. For BAM Friction, all three participants found a different sensitivity of the Gunpowder. For TIL, the order is LANL > IHD > LLNL. For F_{50}, the order is LANL > LLNL (IHD did not test). For ABL Friction, IHD was the only participant to test and found some sensitivity for the Gunpowder. LLNL recorded the least sensitivity of the participants. This has been seen before in the friction testing of other materials by LLNL, and has been attributed to safety shielding of the LLNL equipment[31].

ESD. For ESD all three participants found different levels of sensitivity for Gunpowder. The differences were quite large. The order is LANL > IHD > LLNL. The results by LLNL indicating the Gunpowder is completely insensitive can be explained by LLNL using a custom built system that has a 510-Ω resistor in the circuit. This system is completely different than the ABL ESD systems of LANL and IHD.

Thermal sensitivity. All participants found Gunpowder to have one exothermic transition in the same temperature range. The enthalpy from using a sealed sample holder was higher compared to the enthalpy measured from using a pinhole lid sample holder. This has been seen before in many of the IDCA Proficiency Test materials[2-6].

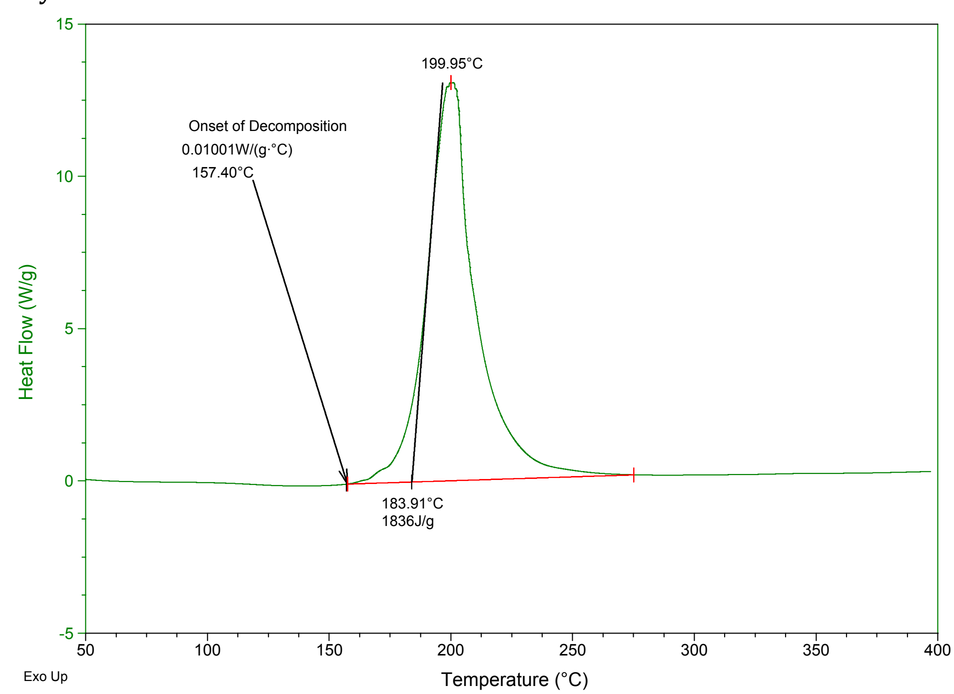

Figure 2. DSC of Gunpowder—LANL Data

Figure 2 shows the DSC profile for Gunpowder taken by LANL. This profile is typical of the profile taken by all the participants in testing, using a pinhole or sealed sample holder.

4.2 Comparison of average values for Gunpowder with standards

Table 9 shows the comparison of the impact, friction and ESD sensitivity of Gunpowder with the standards RDX Type II Class 5 and PETN Class 4.

Impact sensitivity. Because of the wide range of sensitivity values reported, the participants found the Gunpowder to be more or less sensitive than the RDX standard. LLNL found the Gunpowder to be significant less sensitive, LANL found it to be the same sensitivity, and IHD found it to be more sensitive. None of the participants though, found the Gunpowder to be more sensitive than the PETN standard.

Friction sensitivity. All participants found the Gunpowder to be more sensitive than the RDX standard, but less sensitive than PETN standard.

Spark sensitivity. All participants found the Gunpowder to be either the same or less sensitive than RDX. LANL found it to be the same sensitivity as PETN while IHD found it to be more sensitive than PETN. LLNL measured no sensitivity on the custom system with a 510-Ω resistor in the circuit.

Thermal sensitivity. All participants found the Gunpowder to have essentially the same thermal sensitivity, close to the sensitivity of the PETN standard. The T_{max} and ΔH_{exo} for RDX[4] and PETN[12], respectively are: ~ 240°C, ~ 2200 J/g; ~ 205 °C, ~ 1100 J/g.

5 CONCLUSIONS

Conclusions from this study are:

1. Impact testing
    a. The DH_{50} values for Gunpowder varied significantly among participants
    b. LLNL found the Gunpowder much less sensitive, LANL found it to have the same sensitivity, and IHD found it to be more sensitive than the RDX standard
    c. All participants found Gunpowder to be less sensitive than the PETN standard
2. Friction testing
    a. LLNL, LANL and IHD found Gunpowder to be moderately sensitive with BAM friction
    b. IHD found Gunpowder to be moderately sensitive with ABL friction
    c. LLNL, LANL, and IHD found Gunpowder to be more sensitive that the RDX standard and less sensitive than the PETN standard
3. Spark testing
    a. LANL found Gunpowder to be the same sensitivity as the RDX and PETN standards
    b. IHD found Gunpowder to be less sensitive than the RDX standard but more sensitive than the PETN standard
    c. LLNL found Gunpowder to be insensitive
4. Thermal testing
    a. All participants found Gunpowder to have the same sensitivity regardless of sample holder type—pinhole or sealed
    b. All participants found Gunpowder to have about the same thermal sensitivity as PETN and RDX.

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ABREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
DH_{50}	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
ESD	Electrostatic Discharge
F_{50}	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
fps	feet per second
H	Chemical symbol for hydrogen
H_2O	Chemical formulation for water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
j	joules

$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
kg	kilograms
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
N	Chemical symbol for nitrogen
$NaClO_3$	Sodium Chlorate
NSWC	Naval Surface Warfare Center
O	Chemical symbol for oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard Deviation
SEM	Scanning Electron Micrograph
Si	silicon
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

ACKNOWLEDGMENTS

The IDCA thanks Heidi Turner (LLNL) and Gary Hust (LLNL) for supporting data in this document.

This work was performed by the Integrated Data Collection Analysis (IDCA) Program, a five-lab effort supported by Los Alamos National Laboratory, Lawrence Livermore National Laboratory, Sandia National Laboratories, the Air Force Research Laboratory, and Indian Head Division, Naval Surface Warfare under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division. Los Alamos National Laboratory is operated by Los Alamos National Security, LLC, for the United States Department of Energy under Contract DE-AC52-06NA25396. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. This work was performed under the auspices of the U. S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. The Air Force Research Laboratory (AFRL/RXQF) and Indian Head Division, Naval Surface Warfare (NSWC IHD) also performed work in support of this effort. The work performed by AFRL/RXQL and NSWC IHD is under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division.

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